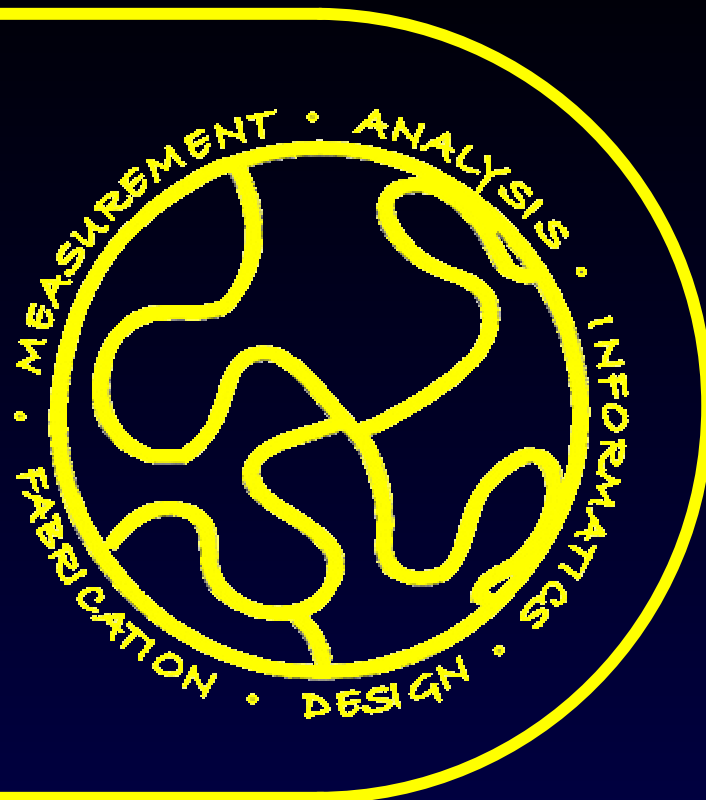


NIST Combinatorial Methods Center

*Accelerating the Pace
of Materials Research*



Michael J. Fasolka
Cher Davis
Alamgir Karim

Project Leader
Technical Coordinator
Group Leader



NIST Combinatorial Methods Center

Two Complimentary Efforts Aimed at Advancing Combinatorial Methods in Industrial, Government and Academic Materials Research:

Research and Development Program

Development of techniques, systems and instrumentation specifically designed to advance the state of the art in combinatorial materials research.

Applications that demonstrate how combi methods are incorporated into real research.

Outreach Program

Conduit for the dissemination of information concerning combinatorial techniques:

- NCMC research products
- Instrumentation designs
- Best practices
- Technique protocols

Mechanism for gauging industrial needs.

NCMC Outreach Program:

Dissemination and Education

- **NCMC Membership Program**

A consortium of institutions interested in developing combinatorial research capabilities

- Tri-annual Member's Workshops
- Direct contact via site visits and visiting scientist collaborations
- Detailed NCMC protocols and instrument specification documents
- NCMC generated automation/analysis software tools
- Database of NIST and Member generated preprints and reprints related to combinatorial materials research
- Limited access Members' Web Site
- Opportunities for deeper collaboration through "Focused Projects"

- **Public Education, Public Relations, Combi Promotion**

- Traditional journal publications, review articles and NIST documents.
- Public presentations and combi lab tours
Nanotech OpenHouse, Nobel Laureate R. Smalley, many others
- Public Web Site (www.nist.gov/combi)
- Meeting Organization



NCMC Membership

As of Sept. 2002



- 13 Members at Participating Member Level (\$10K annual fee)
 - 12 Industrial and 1 Government (AFRL) Partners
 - Some with existing combi capabilities, some with no combi program
- Extensive on site collaborations conducted with Rhodia (K. Krishana) and AFRL staff.
- Current Membership Targets: GE R&D, Academia, Tech./MEMS Firms

Current and Future Development

- **Increase Membership Diversity**

<u>Target</u>	<u>Strategy</u>
Gov't Labs	Demonstrate Productivity Increases
Academia	Demonstrate Combi Knowledge Generation
High Tech Sector	Development of combi methods for a wider range of scientific endeavors.
Instrument Industry	NCCM as a means to gauging trends in combi Fabrication of NCCM instrumentation

- **“Focused Projects” (Level 2 NCCM Membership)**

- Collection of NCCM members fund NIST research directed towards a specific combi problem of common interest.
- NCCM recruits expertise, directs research, and provides early disclosure of (ultimately) publishable work.
- \$25K-\$35K fee per member, depending upon total involved and project cost
- Current Developments: Combinatorial and High-Throughput Measurements of Interfacial Tension (Beers, Hudson, NCCM)

Current and Future Development

- Increasing the value of NCMC Members' Workshops

NCMC-1: Library Design and Calibration (4/26/2002)

- One day event
 - Technical program included NCMC presentations and facilities tours
 - Panel discussion for gauging industrial need
- Focus: Formulations Project*

Member Feedback on NCMC-1 revealed:

- The event was too short overall and did not include time for deeper social/technical interaction between Members and Members/NCMC Staff.
- The presentations were good, but there were too few.
- The facilities tours were not “hands on” enough.

- NCMC “Value Added” Products

A steady stream of combi information Members cannot get elsewhere

- Information superceding that found in publications and workshops.
- Maintains member interest in the Center between workshops and helps in their own justification of Membership fees.



NCMC Member's Workshops

NCMC-2: Adhesion and Mechanical Properties

"Novel Techniques for Combinatorial Materials Testing"

A two day workshop slated for October 7-8 2002

- Invited academic speakers will provide background on traditional adhesion and mechanical measurements through tutorial symposia
- Technical symposia detail NCMC combinatorial techniques and instrumentation designed for adhesion and mechanical testing
 - Multilens Contact Adhesion Testing/Instrumentation
 - Combinatorial Film Delamination Technique
 - Combinatorial Peel Test
 - Combinatorial Failure/Craze Measurements
 - High-Throughput Modulus Measurements
- Laboratory demonstrations of our new techniques
- Poster Session of NCMC and NIST-wide combinatorial materials research
- Presentation highlighting NCMC "Value Added" Products
- Panel discussion and social events provide opportunities to gauge need



NCMC-2, October 7-8, 2002
Adhesion and Mechanical Properties
Bldg. 101 / Lecture Rm. B

Monday, October 7 th , 2002		Tuesday, October 8 th , 2002	
8:00 am	Registration, Bldg. 101 / Lecture Rm. B Coffee & Doughnuts	8:00 am	Poster Session-General Combi Methods (Hallway outside Lecture Rm. B) Karin Beers, Organizer
9:00 am	Welcome and Introductions George Nix	9:00 am	Break (Break Room & Hallway) (120 Handouts Delivered)
9:40 am	Recent Advances in Adhesion Studies using Contact Mechanics Wang Chaozhong, Professor, Department of Chemical Engineering, Director of the Polymer Interface Center, Liaohai University	9:40 am	Coffee & Doughnuts
10:00 am	Coffee Break	9:40 am	Overview on Polymer Coils and Fracture using the Copper Grid Technique Chang Ruo, Chemistry Department & NIST Center for Polymer Surfaces, NIST
10:00 am	Materials Issues and Adhesion Impact on Reliability of Coatings & Interconnects Raghu, Professor, Control Systems Research Chair in Engineering, Department of Mechanical Engineering, University of Texas	10:00 am	Combinatorial Measurements of Polymer Crack Growth using the Copper Grid Test Method Karin Beers
11:00 am	Presentation and Test of NIST Service Life Prediction Facility (Bldg. 225) Diana White	10:00 am	Coffee Break
11:00 am	Lunch (NIST cafeteria, Bldg. 101)	10:00 am	A High-Throughput Test Method for Mechanical Properties of Thin Films Diana Harnisch
11:10 am	Multilens Contact Adhesion Test Method (MCAAT) Al Czekaj	10:45 am	Combinatorial Laboratory Demonstrations Diana Stafford, Organizer
12:00 pm	Combinatorial Adhesion Test Examples Diana Stafford	11:00 am	Lunch (NIST cafeteria, Bldg. 101)
1:40 pm	Coffee Break	1:30 pm	NCMC Practical Knowledge Toolkit Mike Fossaka
2:00 pm	Combinatorial Edge Detection Test for Thin Film Reliability Walter Gilling	1:30 pm	Panel Discussion - Future Members Needs Eric Armit, Polymer Division Chief Hapenhouse, Arkansas, Hapenhouse, Inc. Coffee Service
3:20 pm	Combinatorial Peel Test Method for Adhesion	3:30 pm	Laboratory Demonstrations 2 (Optional)

NCMC “Value Added” Products

Toolbox of Practical Knowledge for Combi Research

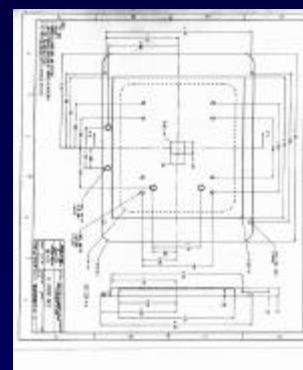
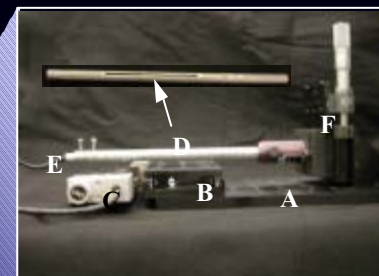
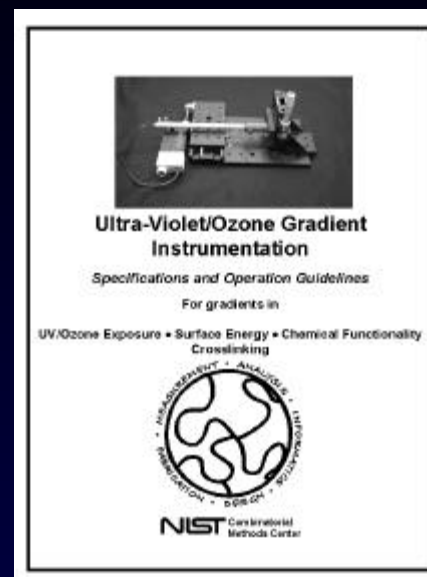
- Instrument Specifications Document Program

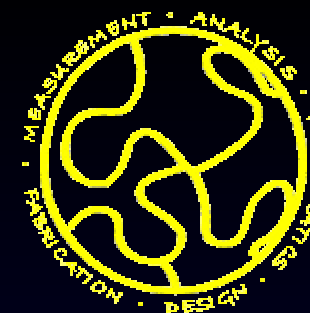
Details not commonly included in journal publications

- Instrument schematics
- CAD/shop drawings
- Detailed component specifications
- Background principles
- Operation guidelines
- Calibration approaches
- Outlines of automation software

Two documents complete, 4 more in production

- Limited distribution: NCMC Members
- *Plans for conversion to NIST publications*





NCMC “Value Added” Products

- NCMC Website (www.nist.gov/combi)



Public site:

- Promotional Materials
- Center Structure, Activities and Capabilities
- Membership Information



Members' Section:

- Password Protected
- A conduit for the dissemination of NCMC research products and information.

Downloadable:

- NCMC-produced Automation and Analysis Software
- NIST/NCMC Combi Publications and *Preprints*
- Presentation Materials from Members' Symposia
- Instrument Specifications Documents

NCMC R&D Program:

Center Research Efforts

- Multivariate Measurement Methods Group Projects
 - *Gradient Techniques*
(some discrete methods)
 - Instrumentation
 - Automation strategies
- NCMC Project
 - New arenas for combi
 - Infrastructure
 - Informatics
 - Collaboration development

Collaboration and Coordination

- Polymers Division
 - Electronic Materials
 - Multiphase Materials
- MSEL
 - Metallurgy, Ceramics
- NIST Wide Efforts
 - BFRL, CSTL, MEL, PL

Many of these relationships grew out of the
Combi Methods Working Group (2000-2002)



Current and Future Development

- **Informatics Infrastructure**

- While many software tools for automation and data analysis have been developed in the MMM group and NCMC, there is no coherent informatics infrastructure to guide our combi “workflow.”
- Members have stated that Informatics issues are a major concern. An accessible example of a working informatics program (however simple at first) would be of great interest.

- **New Arenas of Combinatorial Materials Research**

- Attract a broader range of industries to combi and the NCMC
- Targeting High Tech sector: MEMS, Nanotechnology and Electronics
- Widen NCMC and NIST combi expertise.
- Using current expertise in novel ways.
- Enhance current projects and Facilitate Collaborations (work we've already started!)

Current and Future Development

- **Informatics Infrastructure**

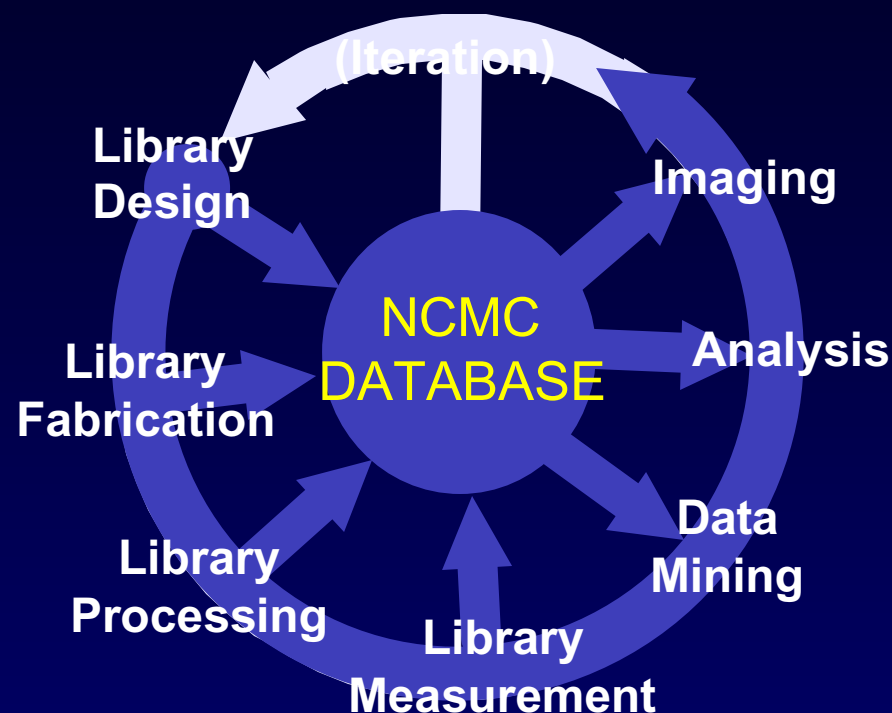
New Guest Researcher dedicated to NCMC infrastructure development:
Dr. Wenhua Zhang (CUNY Staten Island, Dept. of Chem.)

Goal: *Establish an informatics platform that streamlines our combinatorial workflow.*

- **Consolidation/standardization of existing automation and analysis software**

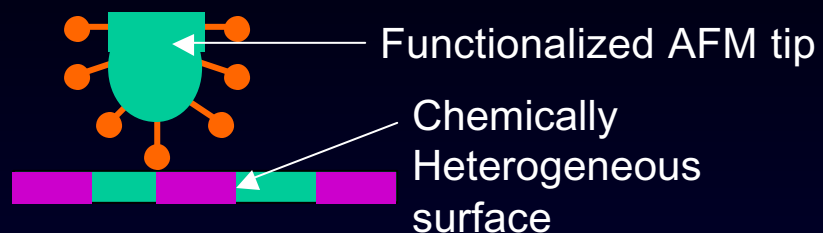
- **Development of a centralized database system with these properties:**

- Open source code (Freely available to NCMC, Members)
- Network based
- Object oriented, Image compatible
- Interfaces with automation tools
- Enables data-mining/versatile analysis of large combi data sets
- E-notebook: Tracks specimen libraries through processing and measurement steps.

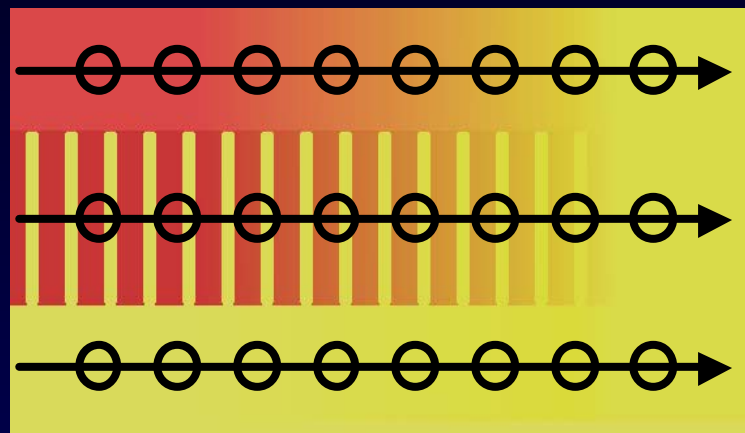


Reference Sample for Chemical Force Microscopy

MSEL contribution to ATP Project with BFRL, PL



- Goal of Project: Move CFM from demonstration stage to industrial technique.
- MSEL contribution leverages (existing!) NCMC expertise in gradient techniques.



Contact <

CFM done here
15 μm pitch lines

Contact <

Pattern with a chemical contrast gradient for testing the sensitivity of CFM.

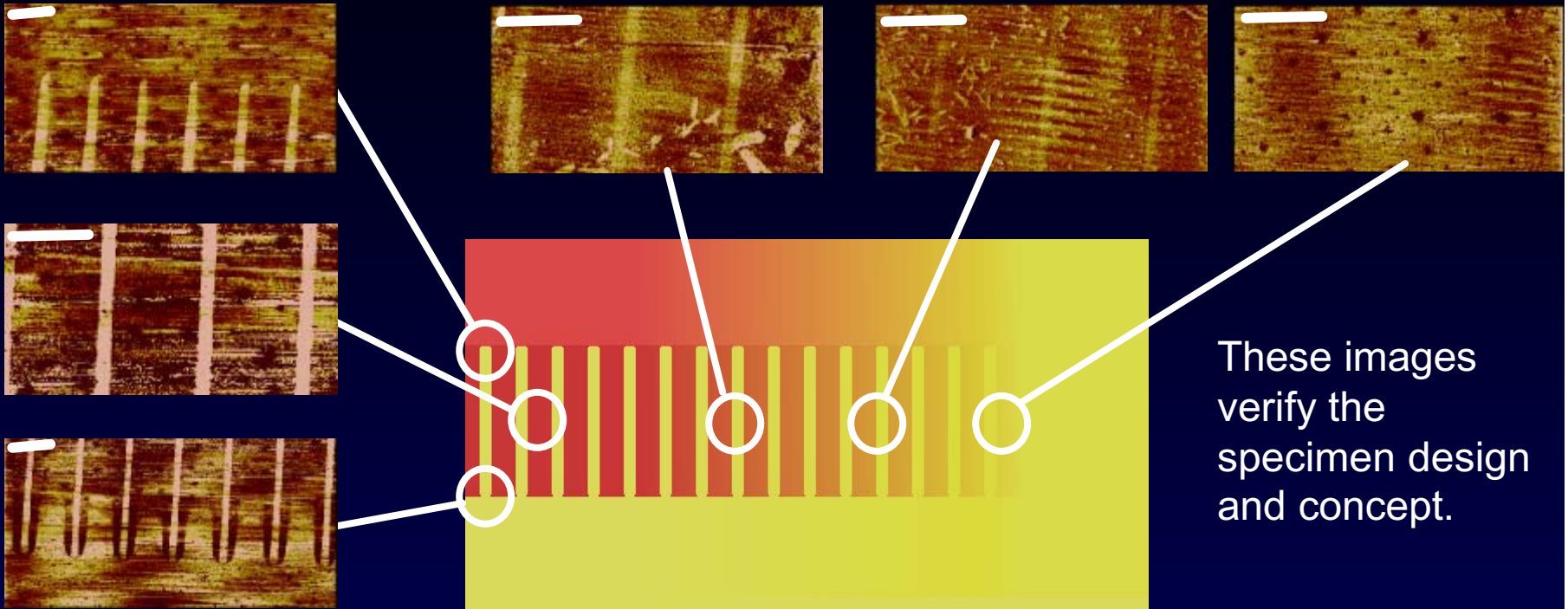
Fabrication and Calibration of Specimen

- **Hydrophilic** and **Hydrophobic** SAMs printed with PDMS stamp
- *Gradient in UV exposure* gradually converts hydrophobic SAM to hydrophilic species.
- Contact < measurements calibrate contrast in the patterned area.

Reference Sample for Gauging the Chemical Sensitivity of CFM

Contact-Mode Friction AFM Images

Scale bars are 10 μ m



These images verify the specimen design and concept.

Challenges:

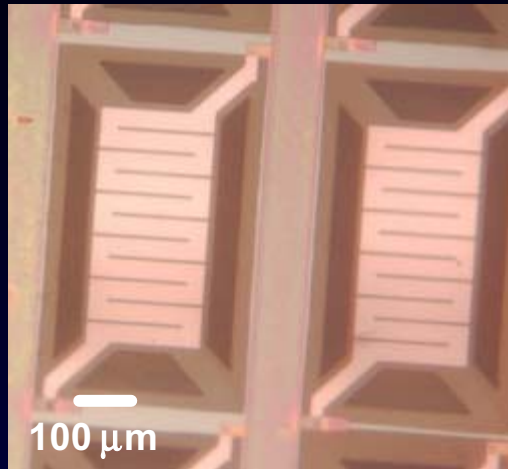
- **Stability:** Current lifetime \approx 3-5 days
Inert Atmosphere, Cleaning?
- **Reproducibility**
Calibration may make this a non-issue

Opportunities

- Substrate for pattern induced film phenomena (e.g. Amit's work)
- SRM or Standard Reference Sample for CFM, Friction AFM, others

Introducing Specimen Libraries to Delicate MEMS

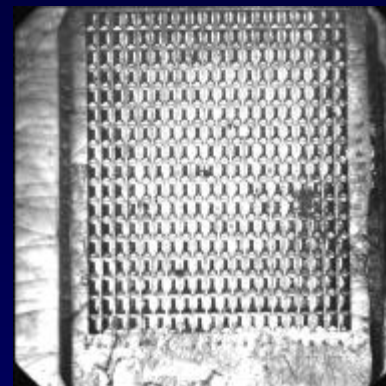
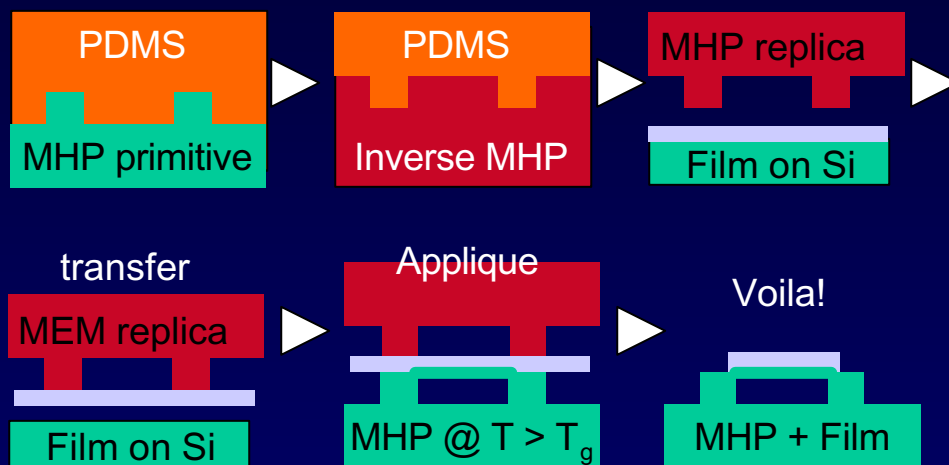
MSEL contribution to ATP Project with BFRL, CSTL



MicroHotplate (MHP) MEMS array (340 elements)

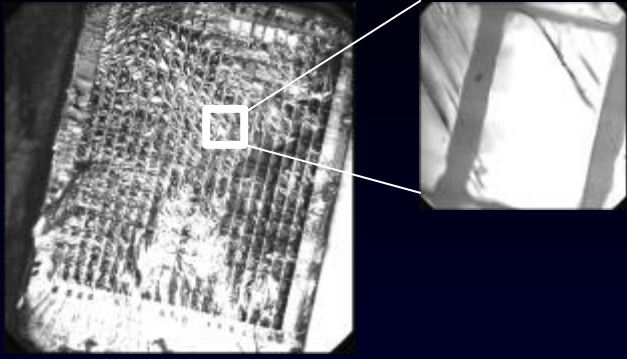
- Goal of Project: Develop combinatorial methods for testing polymer flammability. NIST designed MHP arrays were to be used in this endeavor.
- MSEL contribution is to provide way for depositing films onto (delicate!) MHP MEMs.
An previous attempt to do this through ink-jetting had not produced useful results.

Thermally Induced Film Appliqué



PDMS replica of MHP Array.
Replica introduces free standing film to delicate platforms.

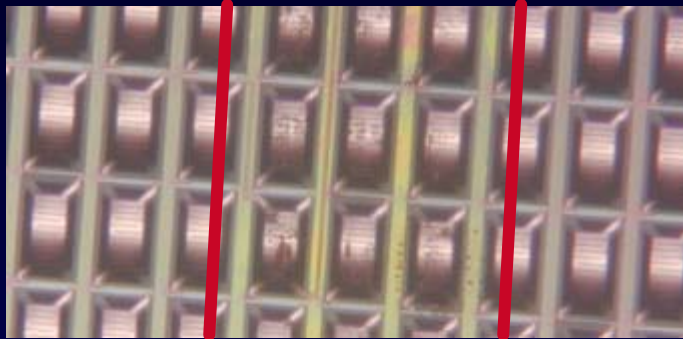
Thermally Induced Film Appliqué



PS-Closite Blend Film Transferred to MHP Replica

- 60nm thick freestanding film suspended on replica structure.
- Very robust; few films punctured

Heated plates



Polymer



No polymer

Demonstration of Selective Film Deposition.

- Since MHPs are addressable, deposition is selective.
- Multiple samples possible via serial deposition or single application of gradient specimens.
- Previous experience (Harrison, Stafford) in transferring films 30nm-1 μ m thick to PDMS slabs.
- With a deposition technique available, MHPs may be an excellent platform for combi-oriented in-situ high temperature AFM.
- Opens the door to MEMS-based Combi

Topographic Feature Height Gradients

with Thom Germer, PL

Combi Tool for Templating and MEMS

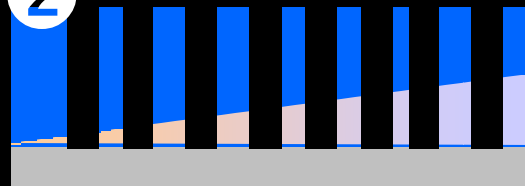
Fabrication Scheme

1



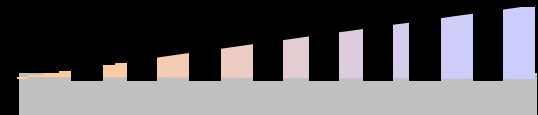
Wedge of Flow Coated Spin-on-Glass, Calcined

2



Lithographic etching via photoresist

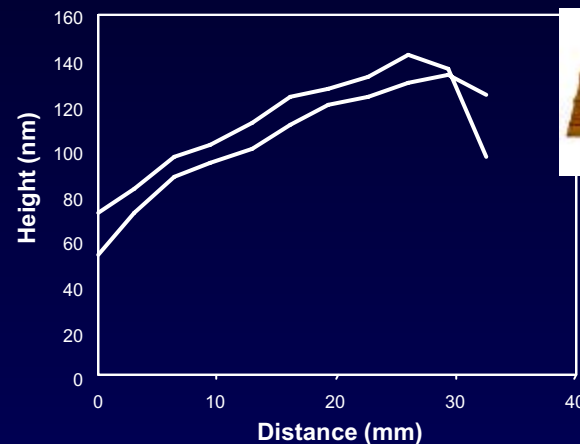
3



Gradient in topographic feature height with designed profile

Demonstration

AFM measurements of feature height gradient. Range is ~100nm.



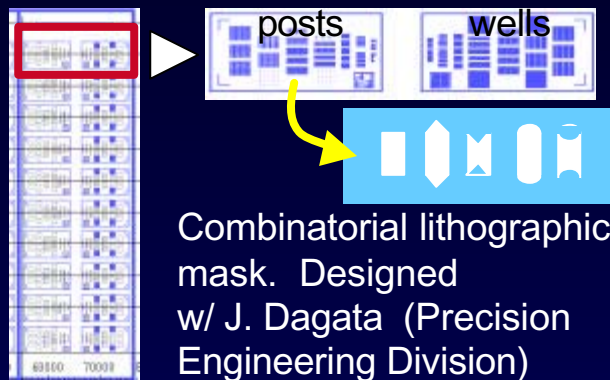
- Topographic effects on film structure are central to templating processes and MEMS device development.
- *Feature height* is a costly and difficult factor to study without gradient technology, as a separate wafer must be created for each h .

Combinatorial Methods for MEMs and Thin Film Devices

Combinatorial tools that foster the development of thin-film opto-electronics, photonic devices, MEMS and nanotechnology.

- Generally, MEMs devices have inherent topographic structure.
- Substrate topography is increasingly recognized as a means of directing and perfecting self-assembled structures for photonics, electronics.
- Combi techniques for examining substrate structure do not yet exist.
- Promise of peripheral technologies, e.g. Tools for soft lithography.

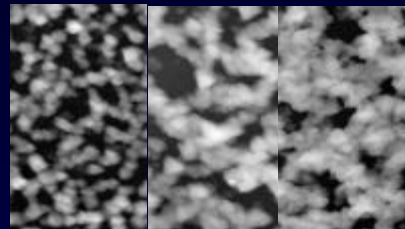
Patterned Topography



Combinatorial lithographic mask. Designed w/ J. Dagata (Precision Engineering Division)

- Spacing, Scale and Shape
- Feature Height Gradients

Micro Roughness Gradients



- Calcined polymer/Spin-on-Glass blends.
- $\text{Roughness} = f(\phi, h)$
- Solution cast via Flow Coating: gradients in ϕ & h

NIST MEMs Technology



Leverage existing tools. Foster NIST relationships.

- Expand MEMs Practice/Use
 - Combi/High Throughput aims
 - Further Develop Deposition
- In-situ Measurements
 - MHP Platform for Hot AFM

FY03: ATP, Competence and Director's Reserve Proposals...

Levels of NCMC Membership

- **Participating Membership**

- Technical meetings presenting work on combinatorial methods from NIST, industry, and others
- Members short courses, workshops, demonstrations
- Members web-site for bulletin board, preprint service, speakers list, etc.

- **Focused Projects**

- Non-proprietary projects on new methods and applications in areas of interest to participants
- Topics chosen by member companies in cooperation with NIST technical teams
- Closed semi-annual meetings; No proprietary information disclosure; All research results publishable
- Administrative support from NCMC

- **Partner memberships**

- CRADA's partnerships with individual member organizations
- Develop methodology and applications to problems of mutual interest
- Projects may involve proprietary information and materials under I.P. agreements
- Coordination support from NCMC